



Template polymerization synthesis of hydrogel and silica composite for sorption of some rare earth elements

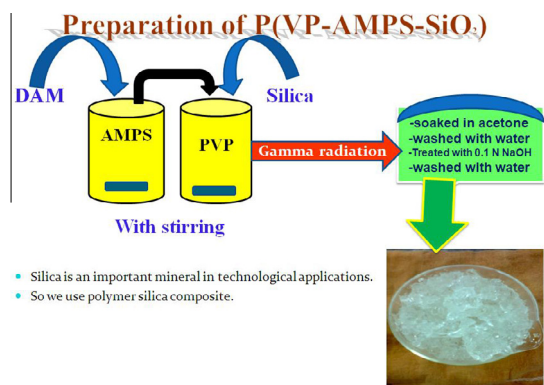


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GRAPHICAL ABSTRACT



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ABSTRACT

New sorbents containing 2-acrylamido 2-methyl propane sulphonic acid monomer onto poly(vinyl pyrrolidone) P(VP-AMPS) hydrogel and P(VP-AMPS-SiO₂) composite have been synthesized by radiation template polymerization. The effect of absorbed dose rate (kGy), crosslinker concentration and polymer/monomer ratio on the degree of template polymerization of P(VP-AMPS) hydrogel was studied. The degree of polymerization was evaluated by the calculated percent conversion and swelling degree. The maximum capacity of P(VP-AMPS) hydrogel toward Cu⁺² metal ion found to be 91 mg/gm. The polymeric composite P(VP-AMPS-SiO₂) has been successfully synthesized. The structure of the prepared hydrogel and composite were confirmed by FTIR, thermal analysis (TGA and DTA) and SEM micrograph. Batch adsorption studies for La³⁺, Ce³⁺, Nd³⁺, Eu³⁺ and Pb⁺² metal ions on the prepared hydrogel and composite were investigated as a function of shaking time, pH and metal ion concentration. The sorption efficiency of the prepared hydrogel and composite toward light rare earth elements (LREEs) are arranged in the order La³⁺ > Ce³⁺ > Nd³⁺ > Eu³⁺. The obtained results demonstrated the superior adsorption capacity of the composite over the polymeric hydrogel. The maximum capacity of the polymeric composite was found to be 116, 103, 92, 76, 74 mg/gm for La³⁺, Ce³⁺, Nd³⁺, Eu³⁺ and Pb²⁺ metal ions respectively.

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1. Introduction

One of the commonly used methods for modifying the surface and bulk properties of polymeric materials is to template

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monomers by irradiation technique known as radiation-induced template. Radiation-induced template method has the advantages such as simplicity, low cost, control over process and adjustment of the materials composition and structure. In addition, this method assures the template of monomers that are difficult to polymerize by conventional methods without residues of initiators and catalyst [1]. Radiation-induced template method is simply based on the irradiation of a base polymer either in the presence of a monomer (simultaneous radiation) or without a monomer (pre-irradiation) to create active sites.

In recent years, organic–inorganic polymer hybrids with a large variety of functionality have been studied intensively. The combination of the two components at a molecular level would provide novel properties that are hardly obtained from conventional organic or inorganic materials. There are different models to obtain silica composite. Contrary to the implementation of Stöber chemistry [2,3] or the microemulsion approach [4,5] the template method offers a very promising alternative route to circumvent the disadvantage of elevated temperatures, large amount of surfactants and/or organic solvents. In addition, the polymeric superstructures can also possess further chemical properties, e.g. for stimuli responsive release or entrapping of molecules.

Composite materials are used today in scientific, technological and manufacturing fields. Composite material is a product comprising a combination of dissimilar constituents. Many investigators have prepared composite sorbents, consisting of inorganic sorbents and organic binding matrices to overcome the limitations of organic resins and inorganic adsorbents [6,7]. Also different inorganic adsorbents such as clay minerals, zeolites, metal hydrates, metal phosphates, and metal oxides were handled in the preparation of composite sorbents.

Lanthanides represent an interesting group of elements which are steadily gaining importance in science and industry. They are now increasingly used in industry, medicine and agriculture. Therefore, the global needs for lanthanides will continue to grow during the next decades [8,9]. In this respect, the present work was oriented to prepare new hydrogel and composite sorbents by gamma radiation technique for effective adsorption of LREs.

2. Experimental

2.1. Materials

Unless otherwise indicated, all materials were of analytical grade and were used without further purification. The lanthanide salts were obtained from Fluka Co. (Switzerland). Bi-distilled water was used for solvation, dissolution, dilution and analytical purposes. De-oxygenated water was used for preparation of hydrogel and composite.

2.2. Measurements

The samples were characterized by FTIR, Hartmann & Braun, Michelson, MB-Series 157 (Canada). Scanning electron microscope (SEM) photographs were recorded by JEOL-JSM 6510 LA (Japan) at accelerating voltage of 20 kV. A computerized UV/Vis double beam spectrophotometer of model T80, PG Instruments Ltd. (England), was used for spectrophotometric determination of metal ions. Thermal analysis was undertaken using a Shimadzu thermogravimetric analyzer model TGA-50 (Tokyo, Japan), a heating rate 10 °C/min, under nitrogen atmosphere (20 ml/min) from room temperature up to 800 °C. A cobalt-60 gamma cell of type MC-20 (Russia), was used as an irradiation source for polymerization at the Cyclotron Project, Inshas, Egypt.

2.3. Preparation of P(VP-AMPS) hydrogel

P(VP-AMPS) resin was prepared by template polymerization of 2-Acrylamido 2-methyl propane sulphonic acid monomer onto poly(vinyl pyrididone) P(VP) as a template polymer in the presence of N,N'-methylene diacrylamide (DAM) as a crosslinker.

About 0.5 g of PVP dissolved in 50 ml bi-distilled water and mixed with 0.6 g of DAM. 10 g of AMPS were dissolved in another 50 ml bi-distilled. The two solutions were mixed using mechanical stirrer for 20 s then transferred into glass ampoules. Nitrogen gas was purged into the ampoules to remove the air from the solutions. The glass ampoules were sealed and then subjected to gamma-irradiation at dose 20 kGy at ambient temperature. After irradiation process, the resin was cut into small discs with a stainless steel scissors. The entire prepared resin was immersed twice in excess bi-distilled water and acetone for 4 h to remove the un-reacted monomers. The prepared resin was treated with 0.1 N NaOH to convert the resin into sodium form then left to dry at room temperature.

2.4. Preparation of P(PVP-AMPS-SiO₂) composite

Poly(PolyAcrylamide-sodium styrene sulphonate-silicon oxide) P(AM-AMPS-SiO₂) composite was prepared by radiation induced polymerization of Polyacrylamide polymer with sodium styrene sulphonate monomers and silicon oxide in de-oxygenated water in the presence of DAM as a crosslinking agent.

About 0.5 g of PVP dissolved in 50 ml bi-distilled water and 0.6 g of DAM, 10 g of AMPS. 0.6 gm of silica was dissolved in 50 ml bi-distilled water. The two solutions were mixed then transferred into glass ampoules and nitrogen gas was purged into the ampoules to remove air from the solutions. The glass ampoules were sealed and then subjected to gamma-irradiation at dose 20 kGy in air at ambient temperature. After irradiation process, the composite was cut into small discs. The entire prepared composite was immersed twice in excess bi-distilled water and acetone for 4 h to remove the un-reacted monomers.

2.5. Sorption studies

The batch experiments of the prepared polymeric hydrogel and composite for the adsorption of La³⁺, Ce³⁺, Nd³⁺, Eu³⁺ and pb²⁺ metal ions from aqueous solution were carried out using a thermostatic water bath shaker at 25 °C for 24 h. Dried samples of each of resin and composite 0.05 gm were stirred in a 10 mL of the aqueous solution. The concentration of the metal ions in solution was determined by computerized UV/Vis double beam spectrophotometer using 4-(Pyridyl-2-azo) resorcinol (PAR) as sensitive coloring reagent. Based on the initial and final metal concentrations, the amount adsorbed (mg/g) and percent uptakes were calculated as follows:

$$q(\text{mg/g}) = \frac{(C_i - C_e)V}{m} \quad (1)$$

$$\% \text{uptake} = \frac{(C_i - C_e)}{C_i} 100 \quad (2)$$

where (q) is the maximum capacity (mg/gm), C_i and C_e are the initial and equilibrium concentrations (mg/L) of metal ions respectively. V is the volume of solution in liter and m is the weight of the resin in gm.

The effect of shaking time was investigated at different period from 5 min to 24 h, at 30 °C, pH = 4 and metal ion concentration 100 mg/L. The effect of pH was tested at different values ranging from 1.0 to 5.0. The initial metal concentration was carried out at various initial metal concentration ranged from 100 to

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